REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1. AGENCY USE ONLY (*Leave blank*)

2. REPORT DATE
11/08/76

3. REPORT TYPE AND DATES COVERED

4. TITLE AND SUBTITLE
DETERMINATION OF DECONTAMINATION CRITERIA, DIMP AND DCPD (U)

5. FUNDING NUMBERS
DAMD 17 75 C 5069

6. AUTHOR(S)
O'DONOVAN, P.

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)
AEROJET ORDNANCE & MANUFACTURING COMPANY
DOWNEY, CA

8. PERFORMING ORGANIZATION REPORT NUMBER
81320R15

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)
FORT DETRICK (FREDERICK, MD.)
FREDERICK, MD

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT
APPROVED FOR PUBLIC RELEASE; DISTRIBUTION IS UNLIMITED

12b. DISTRIBUTION CODE

13. ABSTRACT (*Maximum 200 words*)
THIS IS A PROGRESS REPORT ON AEROJET'S STUDIES OF EXPERIMENTS CURRENTLY UNDERWAY (E.G., PLANT GROWTH & DIMP & DCPD LYSIMETER TESTS). A ROUGH DRAFT COPY OF THE ANNUAL REPORT COVERING ACTIVITIES ON THIS PROGRAM BETWEEN JULY 1975 & SEPTEMBER 1976 HAS BEEN SUBMITTED TO THE PROJECT OFFICE. TWO TYPES OF FULL SCALE LYSIMETER TESTS ARE CONTINUING. THESE TESTS UTILIZE FIVE TYPES OF SOIL. PRELIMINARY DATA ON CHEMICAL ANALYSES FROM THE SOIL CULTURE EXPERIMENTS IN WHICH ALFALFA, BEAN, SUGAR BEET, CARROT, & WHEAT PLANTS WERE EXPOSED TO 0, 1, 8 OR 20 PPM DIMP OR DCPD IN IRRIGATION WATER, ACCORDING TO PLAN, HAVE BEEN REPORTED IN THE ANNUAL REPORT.

14. SUBJECT TERMS
CONTAMINANTS, FLORA, SOIL, CHEMICALS, LYSIMETER

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT
UNCLASSIFIED

18. SECURITY CLASSIFICATION OF THIS PAGE

19. SECURITY CLASSIFICATION OF ABSTRACT

20. LIMITATION OF ABSTRACT

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original

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AERÓJET ORDNANCE AND MANUFACTURING COMPANY
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DETERMINATION OF DECONTAMINATION CRITERIA

DIMP AND DCPD (U)

Report No. 1953-01(15)MP

Contract DAMD-17-75-C-5069

to

U.S.ARMY, Ft. Detrick
Fredrick, Maryland 21701

Prepared by: P.A. O'Donovan
P.A.O'Donovan

Date: 8 November 1976

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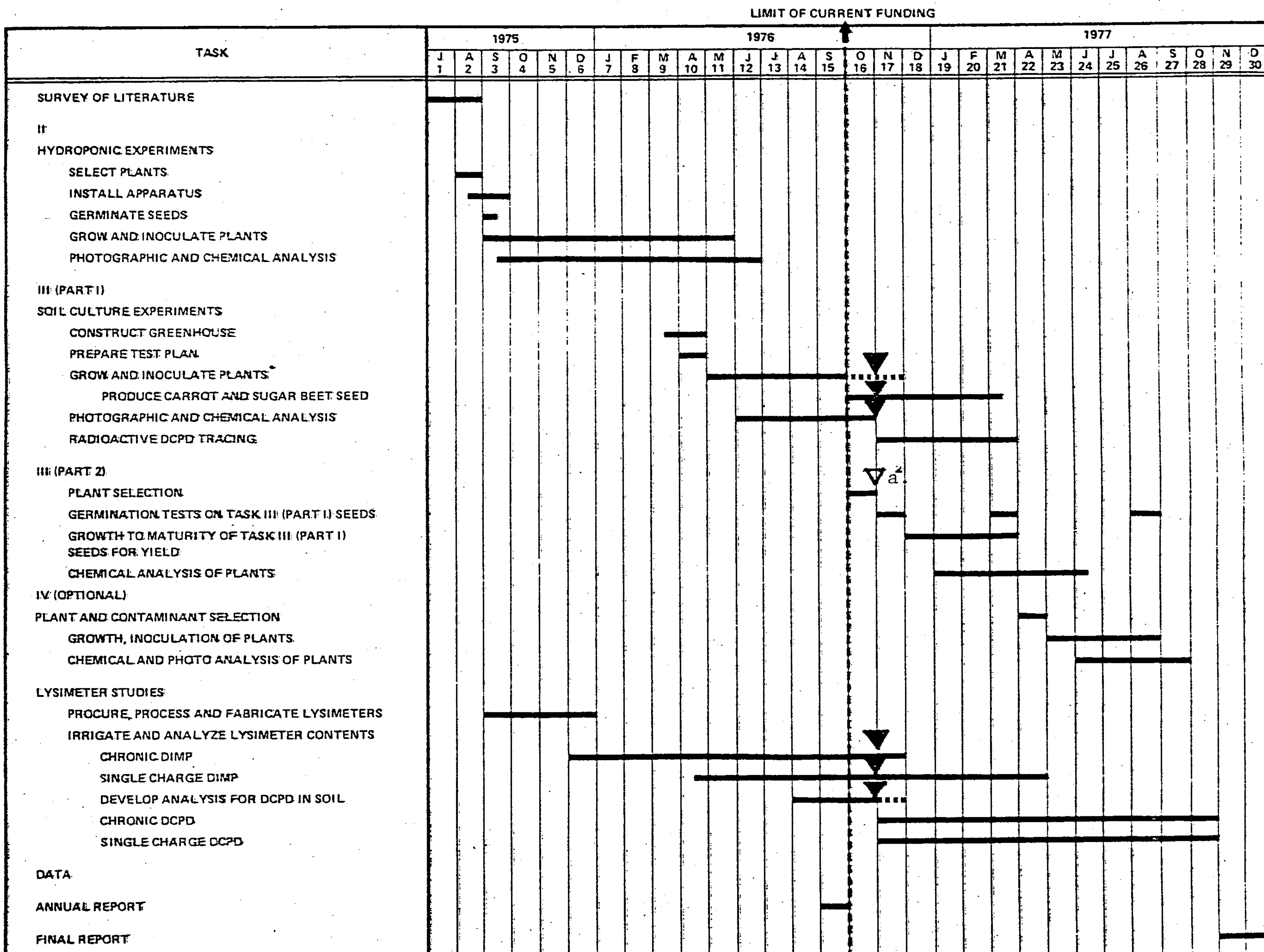
DETERMINATION OF DECONTAMINATION CRITERIA DIMP AND DCPD RESEARCH TASK SCHEDULE



*POSSIBLE SLIPPAGE POINT. ADJUSTMENT OF CONTAMINANT AT THIS POINT SHIFTS ALL FOLLOWING PLANT WORK TO THE RIGHT.

▼ Satisfactory Progress- On Schedule

▽ Slippage of Schedule

a. Task may be eliminated due to reduced level of funding.

Progress on items proposed for action during October, 1976 is discussed in the following paragraphs.

ANNUAL REPORT

A rough draft copy of the annual report covering activities on this program between July 1975 and September 1976 has been submitted to the project office.

FULL SCALE LYSIMETER TESTS

Two types of full scale lysimeter tests are continuing. These tests utilize five different types of soil including:

Chino	-	sandy clay loam
Brawley	-	silty clay
Ventura	-	clay loam
Fullerton	-	sandy loam
Walnut	-	clay loam

The first test vehicle consists of five each, five foot deep reconstructed cylinders of soil contained in epoxy coated steel lysimeters. Each of these lysimeters is irrigated at regular intervals, currently every two weeks, with two inches (12,887 ml) of distilled water containing 20 parts per million (ppm) diisopropyl methyl phosphonate (DIMP). The water percolating down through the soil is sampled at six levels of depth by applying a light vacuum to porous ceramic tensiometers embedded in the soil and by trapping the drainage at the bottom of the lysimeter.

Soil samples are taken in six inch incremental depths by means of a core sampler. This experimental group is designated Group 1 and the most recent data from the chemical analysis of Group 1 water and soil samples are shown in Tables 1 and 2 respectively.

The second group of lysimeters is similar in all respects to the first except that the DIMP was mixed intimately with the top one foot layer of soil to a concentration of 20 ppm and the regular irrigation is made with distilled water. These lysimeters are designated as Group 2 and are sampled and analyzed in the same manner as Group 1. Recent data from the analyses of these samples is tabulated in Tables 3 and 4.

The previously noted characteristics of the analytical data continue to partially hold. That is,the DIMP content in Group 1 generally appears higher with proximity to the surface but there has been a slight decrease in the DIMP concentration from the September values. The Ventura sample is again the exception in Group 1 tensiometer samples.

In Group 2 the data indicates that the DIMP has located itself in a broad band generally through the lower center of the lysimeters.

The DIMP content of the drainage water samples, designated the 60 inch samples, is plotted versus irrigation time in Figures 1a through 1e. It would be expected that the drain sample, being relatively large (ca. 25-50% of the applied water), would be

Table 1

DIMP Content o. Tensiometer Water Samples (Group 1 East)

Depth	Ventura	Chino	Fullerton	Walnut	Brawley
		(ppm @ 282	days)		
6"	9.8	14.7	26.1	16.8	20.8
18"	5.6	15.0	21.2	14.3	17.0
30"	5.4	21.1	17.3	13.1	19.5
42"	4.4	15.8	9.9	12.3	11.6
54"	11.4	15.4	10.1	10.9	14.1
60"	15.2	12.3	10.4	11.7	17.0
		(ppm @ 312	days)		
6"	6.0	21.5	67.3	30.4	29.0
18"	4.6	16.6	31.2	22.6	23.6
30"	3.8	21.3	32.0	18.3	16.6
42"	7.0	14.6	15.8	26.5	16.0
54"	7.7	16.6	5.8	23.6	12.3
60"	15.4	14.1	10.1	15.4	10.5

Table 2

DIMP Content of Group 1 Soils at Sampling Time

Level	Chino	Brawley	Ventura	Fullerton	Walnut
		@ 275 days			
0 - 1/8"	14.0	8.8	12.6	7.1	9.7
1/8" - 6"	8.0	6.1	4.1	4.7	4.0
6" - 12"	6.5	3.2	2.0	4.8	4.0
12" - 18"	6.4	7.7	1.3	5.6	3.4
18" - 24"	5.4	4.6	0.9	4.0	3.0
24" - 30"	5.8	5.4	1.0	4.2	3.7
30" - 36"	3.5	5.6	1.5	4.0	3.6
36" - 42"	2.2	4.4	1.4	3.3	2.3
42" - 48"	1.0	5.7	1.7	4.6	3.4
48" - 54"	2.6	5.6	1.5	2.3	2.7
54" - 60"	4.6	6.9	1.9	4.4	5.1
		@ 303 days			
0 - 1/8"	14.6	4.6	13.6	21.3	9.6
1/8" - 6"	4.7	3.1	1.1	6.4	6.2
6" - 12"	5.9	4.4	0.9	6.3	4.9
12" - 18"	5.4	5.5	*	5.6	3.5
18" - 24"	4.4	4.4	*	5.7	2.6
24" - 30"	5.2	2.9	*	5.9	4.5
30" - 36"	3.2	4.2	0.4	1.1	4.2
36" - 42"	3.9	3.4	*	2.7	2.5
42" - 48"	3.9	4.5	*	3.3	4.5
48" - 54"	3.6	3.9	*	3.6	4.0
54" - 60"	4.4	4.1	*	2.5	5.5

* <0.1 ppm

Table 3

DIMP Content of Tensiometer Water Samples (Group 2-West)

Depth	Ventura	Chino	Fullerton	Walnut	Brawley
		(ppm @ 147	days)		
6"	1.1	1.8	1.2	1.4	1.5
18"	15.7	131.6	16.5	0.6	26.2
30"	24.9	33.8	19.5	42.7	19.4
42"	6.5	-	-	35.3	*
54"	0.9	0.9	8.2	*	4.4
60"	0.4	*	0.3	*	0.7
		(ppm @ 161	days)		
6"	*	*	*	*	2.0
18"	11.3	42.1	13.5	0.6	28.6
30"	163.3	43.7	26.8	33.6	22.1
42"	11.3	13.2	24.6	110.0	*
54"	*	1.5	7.5	*	*
60"	*	*	*	*	*

*< 0.1 ppm

Table 4

DIMP Content of Group 2 Soils at Sampling Time

Level	Chino	Brawley	Ventura	Fullerton	Walnut
		@ 169 days			
0 - 1/8"	*	*	*	*	*
1/8" - 6"	*	*	*	*	*
6" - 12"	*	*	*	*	*
12" - 18"	*	*	*	*	*
18" - 24"	6.3	6.5	*	2.4	*
24" - 30"	14.4	31.3	6.5	2.7	4.0
30" - 36"	19.5	16.5	22.4	1.2	11.2
36" - 42"	15.4	*	27.0	2.5	23.2
42" - 48"	2.6	*	14.8	4.4	18.7
48" - 54"	*	*	4.2	10.7	2.8
54" - 60"	*	*	*	14.4	*
Drain H_2O			10-13-76 2.2	10-6-76 2.5	

* <0.1 ppm

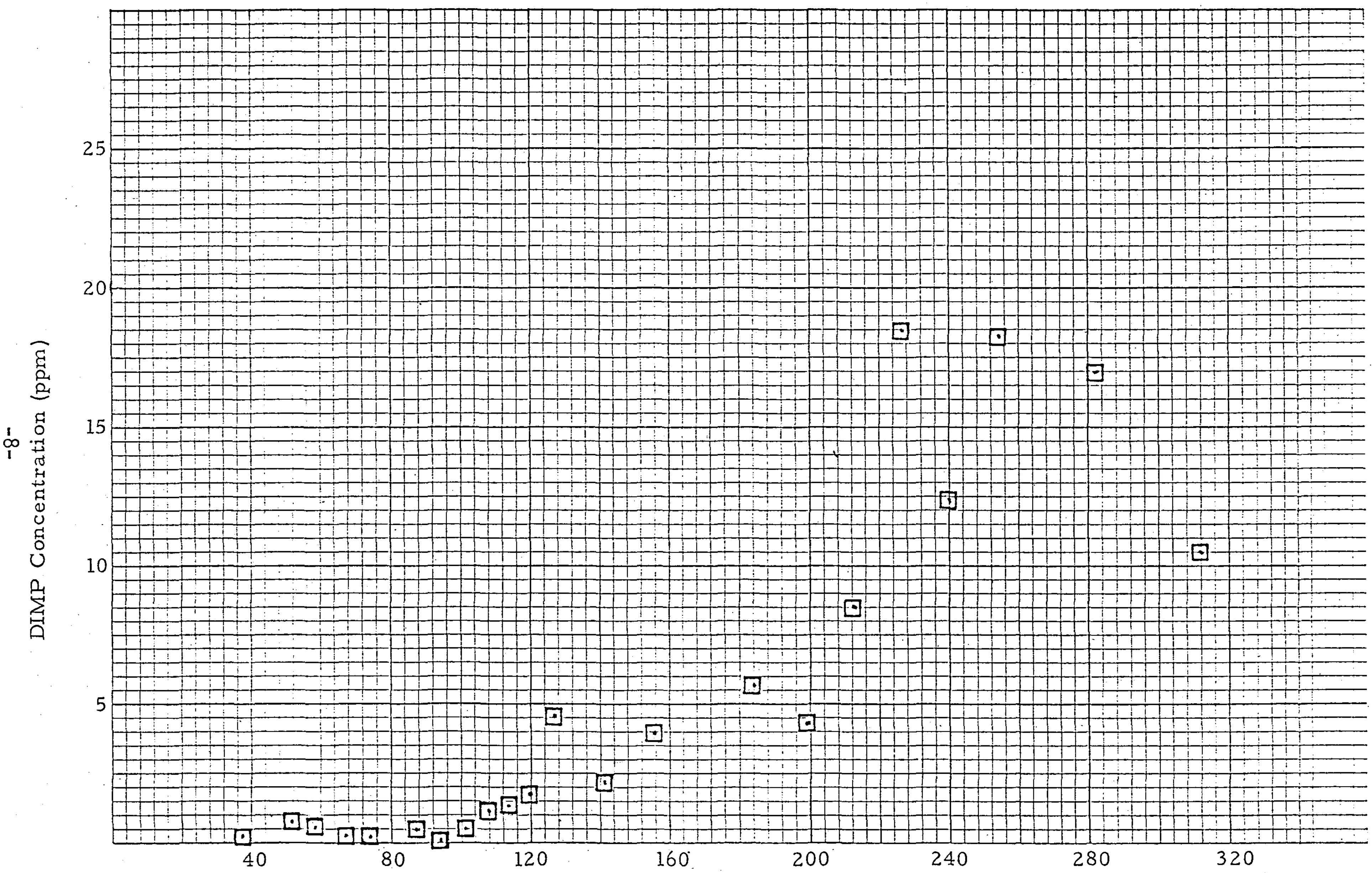


Figure 1(a) Concentration of DIMP in 60 inch Sample of Water

Brawley Lysimeter

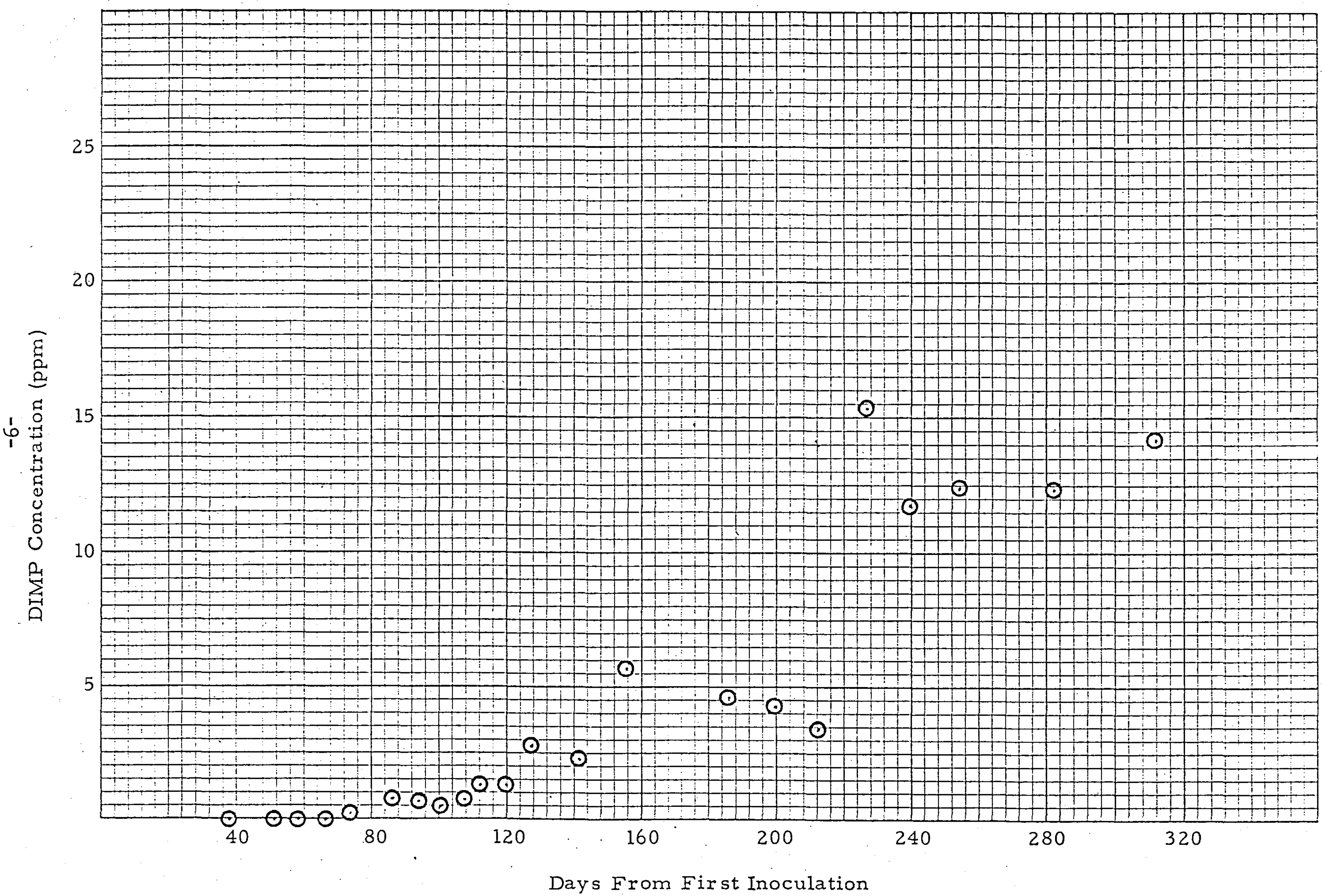


Figure 1(b) Concentration of DIMP in 60 inch Sample of Water

Chino Lysimeter

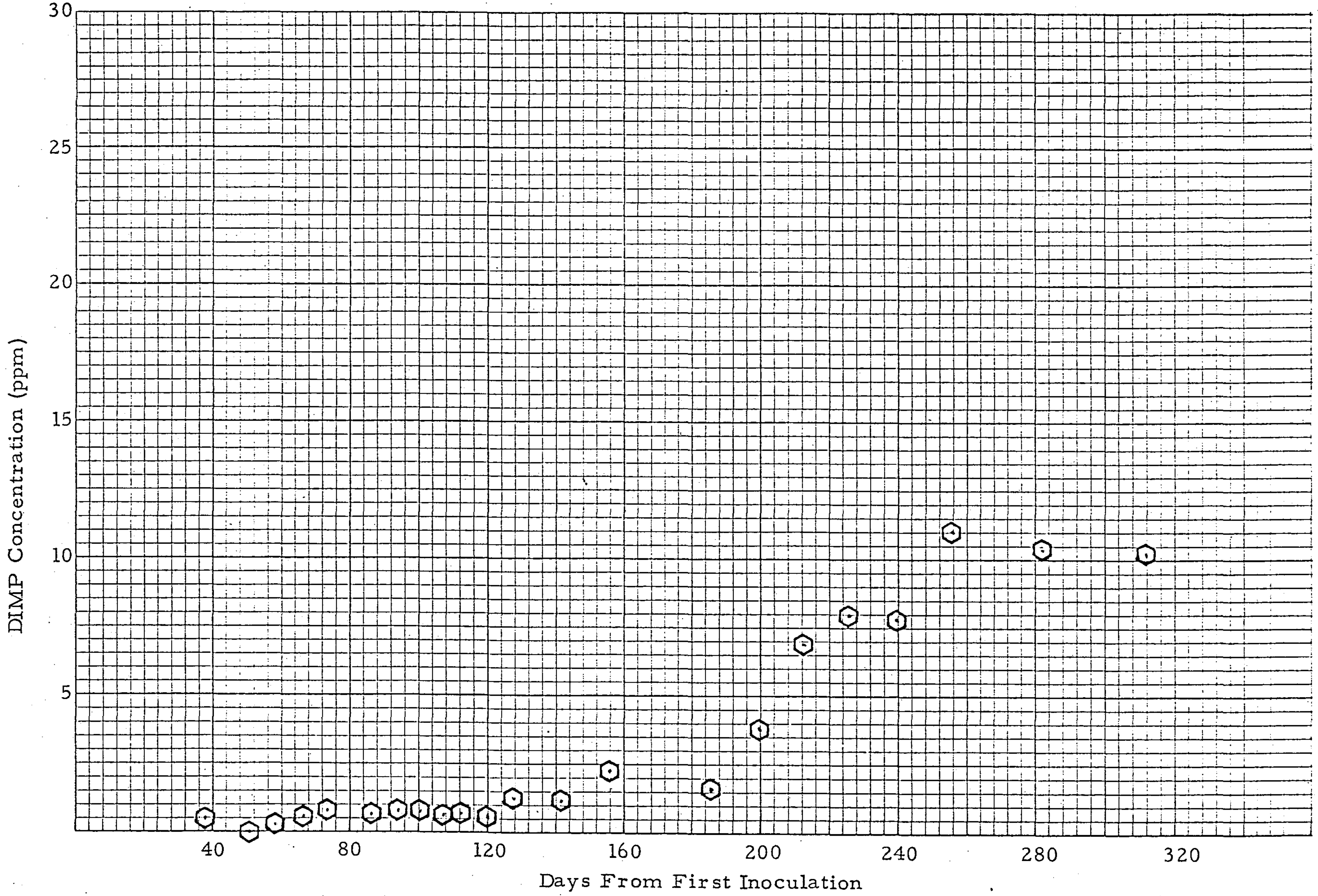


Figure 1(c) Concentration of DIMP in 60 inch Sample of Water
Fullerton Lysimeter

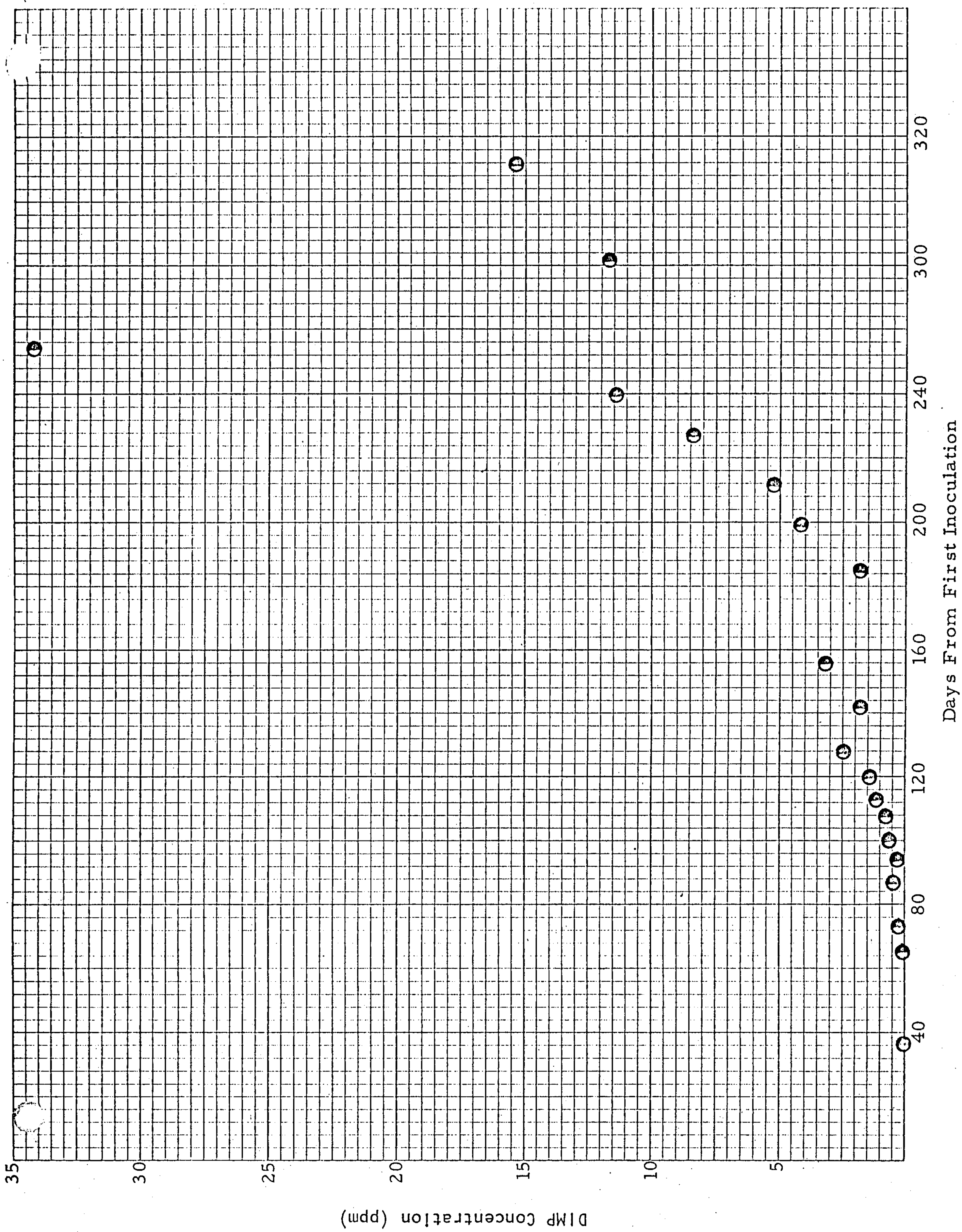


Figure 1(d) Concentration of DIMP in 60 inch Sample of Water Walnut Lysimeter

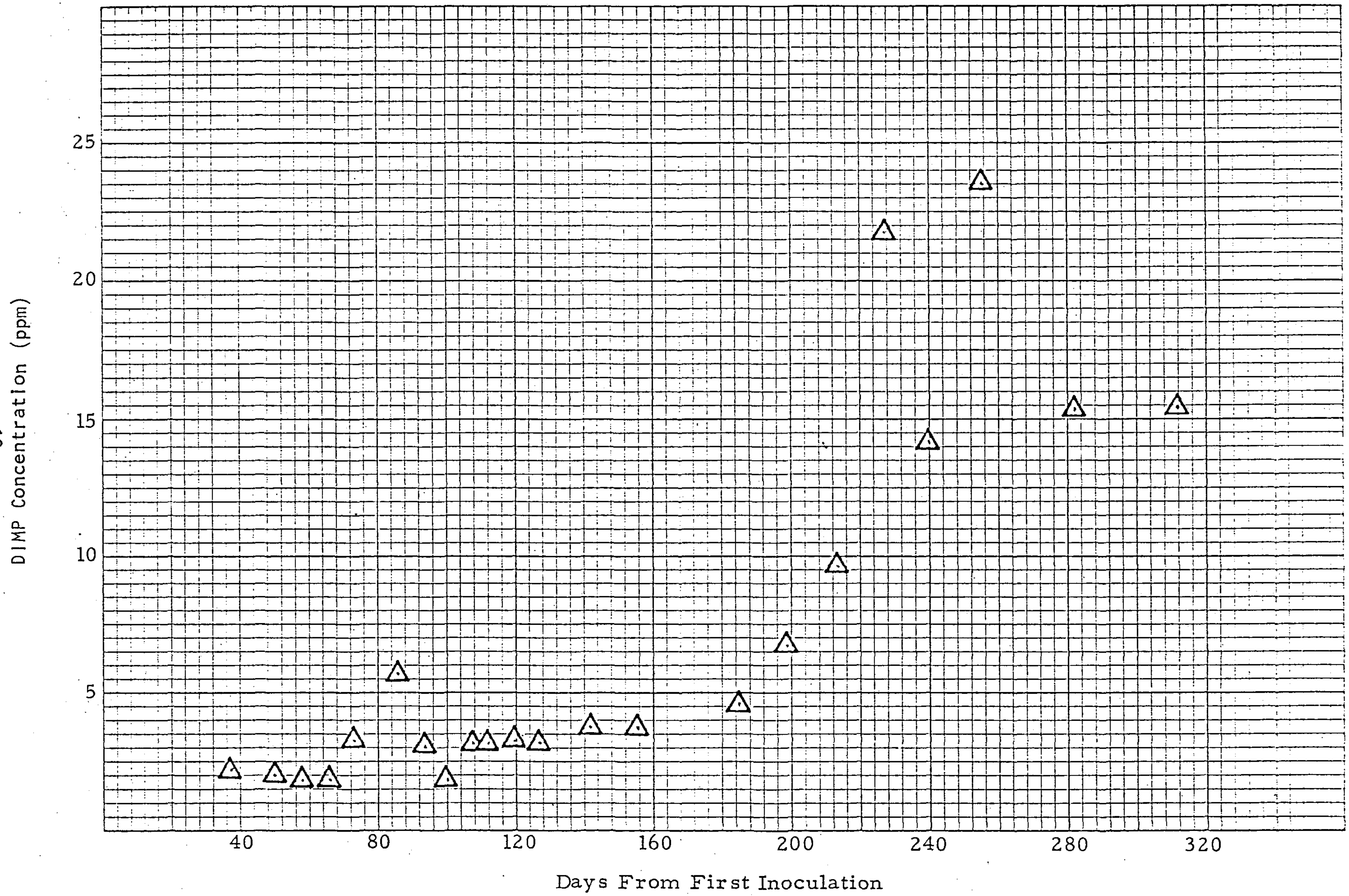


Figure 1(e) Concentration of DIMP in 60 inch Sample of Water
Venture Lysimeter

truly representative of the 60 inch depth and not suffer from the inadequacies of the tensiometer samples which are relatively small. It would also be anticipated that after all adsorptive equilibria have been satisfied the addition of 20 ppm DIMP in the irrigation water to the surface of the lysimeters would result in a concentration in the drain water of 20 ppm DIMP. Figures 1a through 1e indicated that the drain (60 inch) samples may be approaching this condition.

The fraction of the volume of water applied to the surface of the lysimeters that exits at the drain has been termed drainage ratio. This is indication of the amount of water evaporated from the surface and/or bound in some way to prevent its exiting the drain. The Group 1 data in Figure 2 are averages of pairs of successive drain water recoveries. The Group 2 data in Figure 3 is a plot of all of the data points from that group. All of the data points from Figures 2 and 3 are averaged for each group at each time interval and presented in Figure 4.

SOIL CULTURE EXPERIMENTS

Preliminary data on chemical analyses from the soil culture experiments in which alfalfa, bean, sugar beet, carrot and wheat plants were exposed to 0, 1, 8 or 20 ppm DIMP or DCPD in irrigation water according to the test plan in Report No. 1953-01(11)MP have been reported in the annual report. The alfalfa, wheat and beans have been harvested, weighed, and held in the freezer for future analysis and/or disposition. The carrots and sugar beets are about ready to harvest. The bio-mass data from all five species will

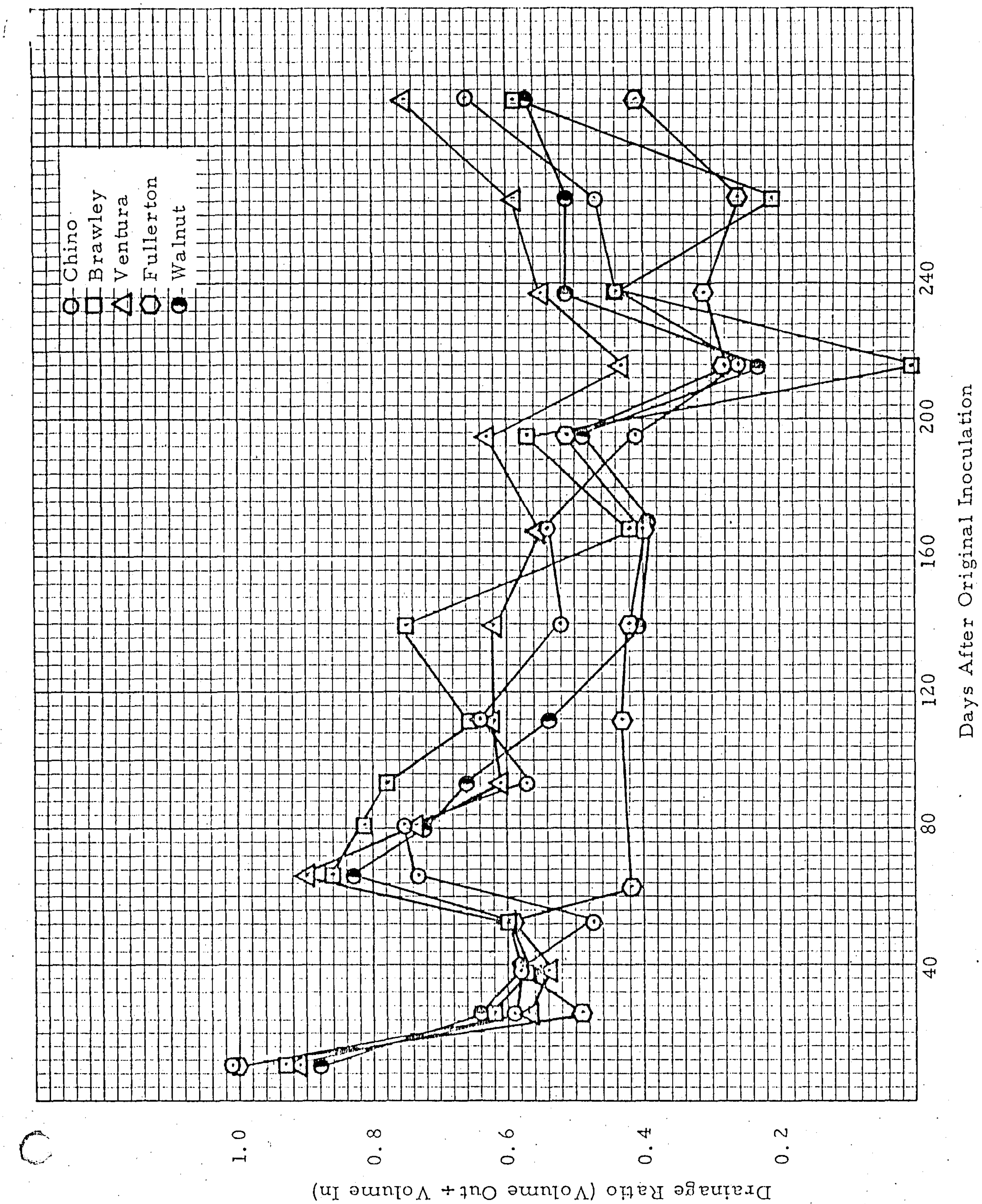


Figure 2., Drainage Ratios of Various Soils in Full Scale Lysimeter

Group 1

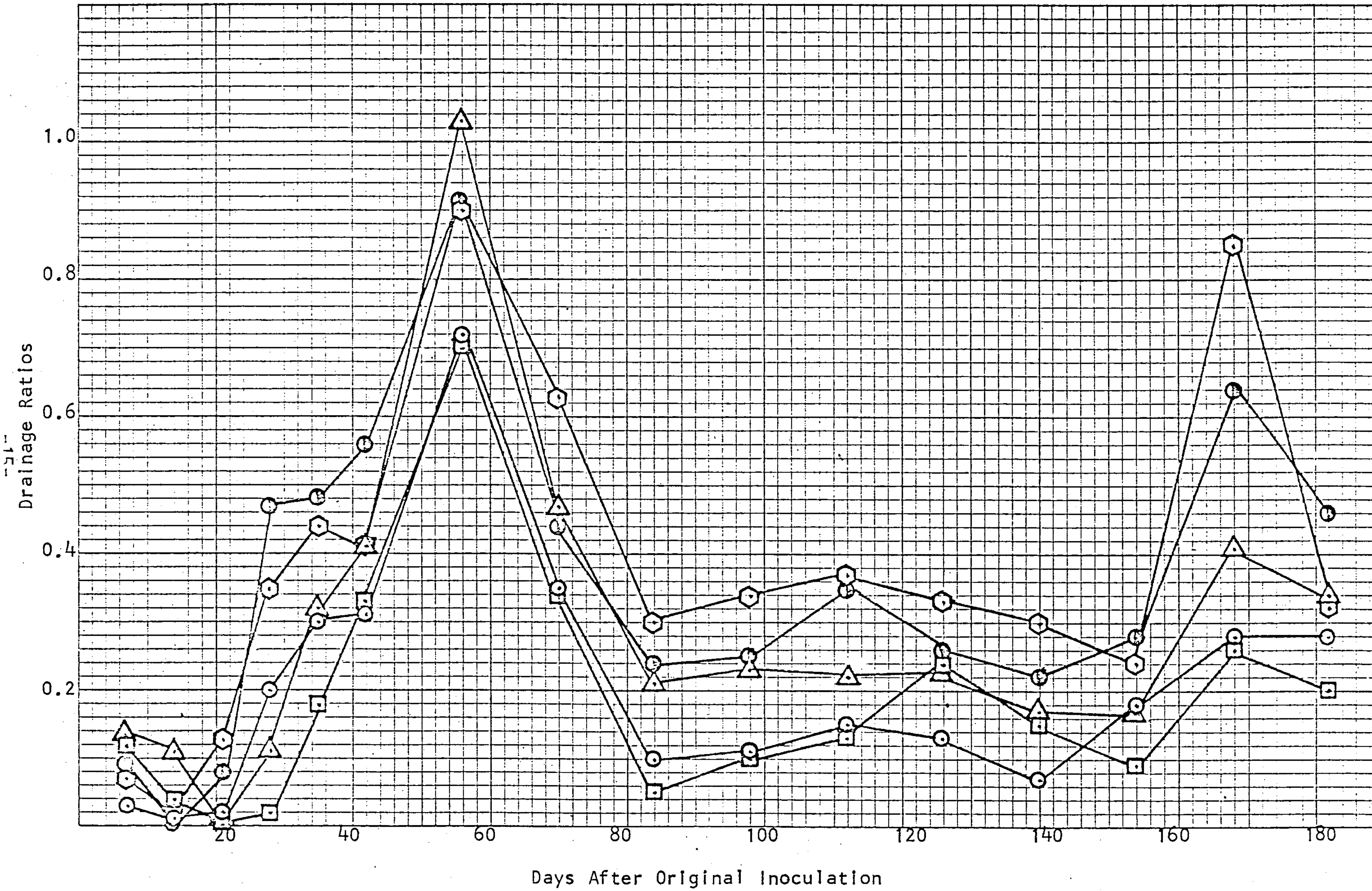


Figure 3. Drainage Ratios of Various Soils In Full Scale Lysimeters

Group 2

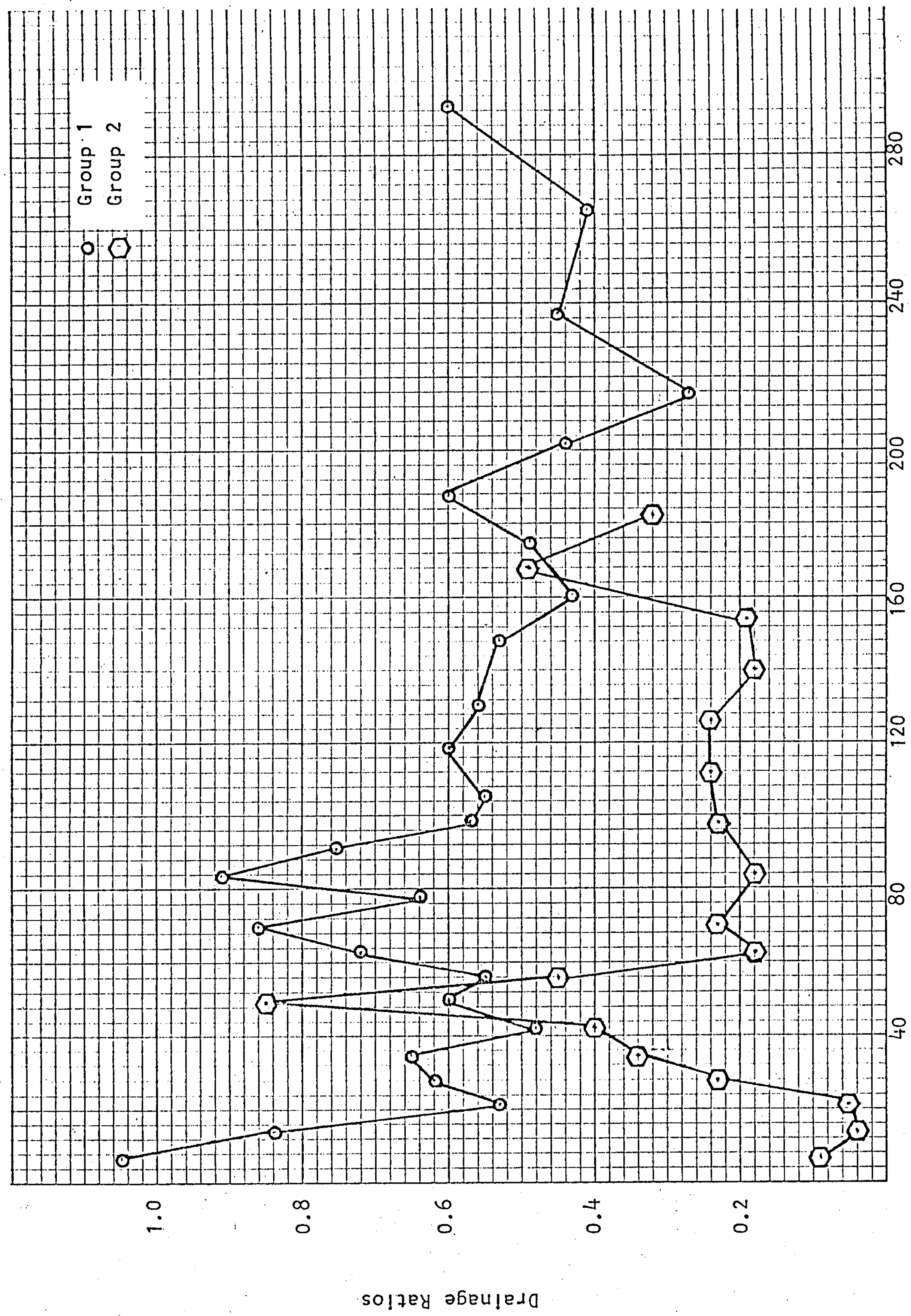


Figure 4. Drainage Ratios of Various Soils in Full Scale Lysimeters

Average of All Samples Within the Groups

be subjected to statistical analysis to determine the effective significance of these contamination levels. Visual examination of these plants has shown no symptoms of phytotoxicity which can be attributed to either the DIMP or DCPD. The statistical data will be presented at a future time subsequent to the carrot and sugar beet harvest.

The range finding tests being run in soil to determine gross effective levels are continuing. As of this writing the exposure has been continued for 90 days to 0, 50, 100, 300, 500, 700 and 1000 ppm DIMP and DCPD. To those plants which originally germinated the DCPD exposures at all levels appear to be ineffective. The DIMP exposures, on the other hand, appear to have an effect at all levels, 50 ppm irrigation having minimal symptoms of leaf burn in the carrot and alfalfa and excessive leaf curl in the sugar beet. From the 100 ppm DIMP irrigation and up there is severe damage to all the plants. The 50 ppm. plants have so far been exposed to a total of 15,600 ml. of irrigation water containing 780 milligrams of chemical contaminant.

DCPD ANALYSIS IN SOILS

One of the difficulties encountered in the analysis of soil for DCPD content has been the inability to recover quantitatively DCPD standards added to soil samples. Recoveries have varied from 18 to 80 percent of theoretical. Various solvents have been tried as extraction media with little success.

Subsequent to the site visit by the Project Officer and other project management personnel a series of tests was initiated to investigate this problem. The initial approach consists of weighing a known amount of DCPD standard into a small amount of screened, air dried soil (ca.5.g) and incorporating this mixture into a larger (ca.500g.) quantity of soil by tumbling them together in a sealed jar. This becomes the standard sample from which dilutions are made and samples taken. Analysis of these samples is made by extracting the DCPD from the soil with agitation in a known volume of solvent, clarifying by centrifugation and chromatographing (gas liquid chromatography) the resulting solution.

At first a series of standard solutions were prepared from DCPD dissolved in hexane ranging from 3.4 ppm to 340 ppm DCPD. Chromatographing on a Varian Model 1840-1 gas-liquid chromatograph fitted with a flame ionization detector followed by peak integration by the cut and weigh method gave the data shown in Table 5. The column used was 12' x 1/8", 10% QF-1 on gas chrom Q.

The chromatographic response shown in Table 5 should be sufficient to enable determination of extraction efficiencies in the soil system. The first sample to be subjected to hexane extraction was the 340 ppm DCPD in soil. Eight samples of 5 grams each were taken from a batch of 450 grams of the contaminated soil which had been tumbled on a laboratory ball mill drive for ten hours. These eight samples were extracted by agitation in 5 ml. hexane each and the hexane later chromatographed. The average recovery

TABLE 5

CHROMATOGRAPHIC RESPONSE TO DCPD

Sample No.	Conc. (ppm.)	Sample Vol.	Attenuation	Corrected Peak Weight (ppm.)
D	3.4	1.8	4×10^{-11}	66
"	"	1.9	"	63
"	"	1.8	"	98
"	"	1.9	"	63
C	34	1.9	"	55
"	"	2.0	"	58
"	"	1.9	"	66
"	"	1.8	"	66
B	170	1.7	16×10^{-11}	68
"	"	1.8	"	66
"	"	1.8	"	61
A	340	0.9	"	61
"	"	1.0	"	48
"	"	1.0	"	60

Average = 64.8

Std. Deviation = ±10.5 = ±16.2%

of initially added DCPD was 178 ppm or 52%. Distilled water, pH 9.0 buffer and pH 4.0 buffer were also used as extraction liquids on similar soil samples. Integration of these curves is not complete but qualitatively it can be seen that in all three conditions; neutral, acid and base, there is less DCPD than in the case of hexane.

It has been assumed that volatility of the DCPD could be the limiting problem in the analytical procedure. To ascertain the significance of this phenomenon several limited experiments have been initiated. The first consisted in placing a series of 5 gram samples of the 340 ppm DCPD-in-soil samples in open Petri dishes on the laboratory bench, extracting them with hexane at selected time intervals and chromatographing the extracts. These samples were from the same container which previously yielded an average of 52% DCPD recovery on hexane extraction. This later test yielded the data in Table 6.

Plotting the data in Table 6 gives the curve in Figure 5. Similar curves are being prepared for more dilute DCPD in soil samples.

It it can be shown by experiments such as the above along with an independent determination of a high extraction efficiency solvent then a reassessment of the proposed DCPD technique for lysimeter experiments will be required.

PROPOSED ACTIVITY DURING NOVEMBER 1976

- Revise the Research Task Schedule to conform to new funding and task emphasis requirements.

TABLE 6

EVAPORATION OF DCPD FROM EXPOSED SOIL SAMPLES

Sample No.	Exposure Time (min)	DCPD Found (ppm)	%
1	~0	134	39
2	80	26	7.6
3	145	18	5.3
4	220	16	4.7
5	270	14	4.1
6	1350	< 0.1	< 0.03

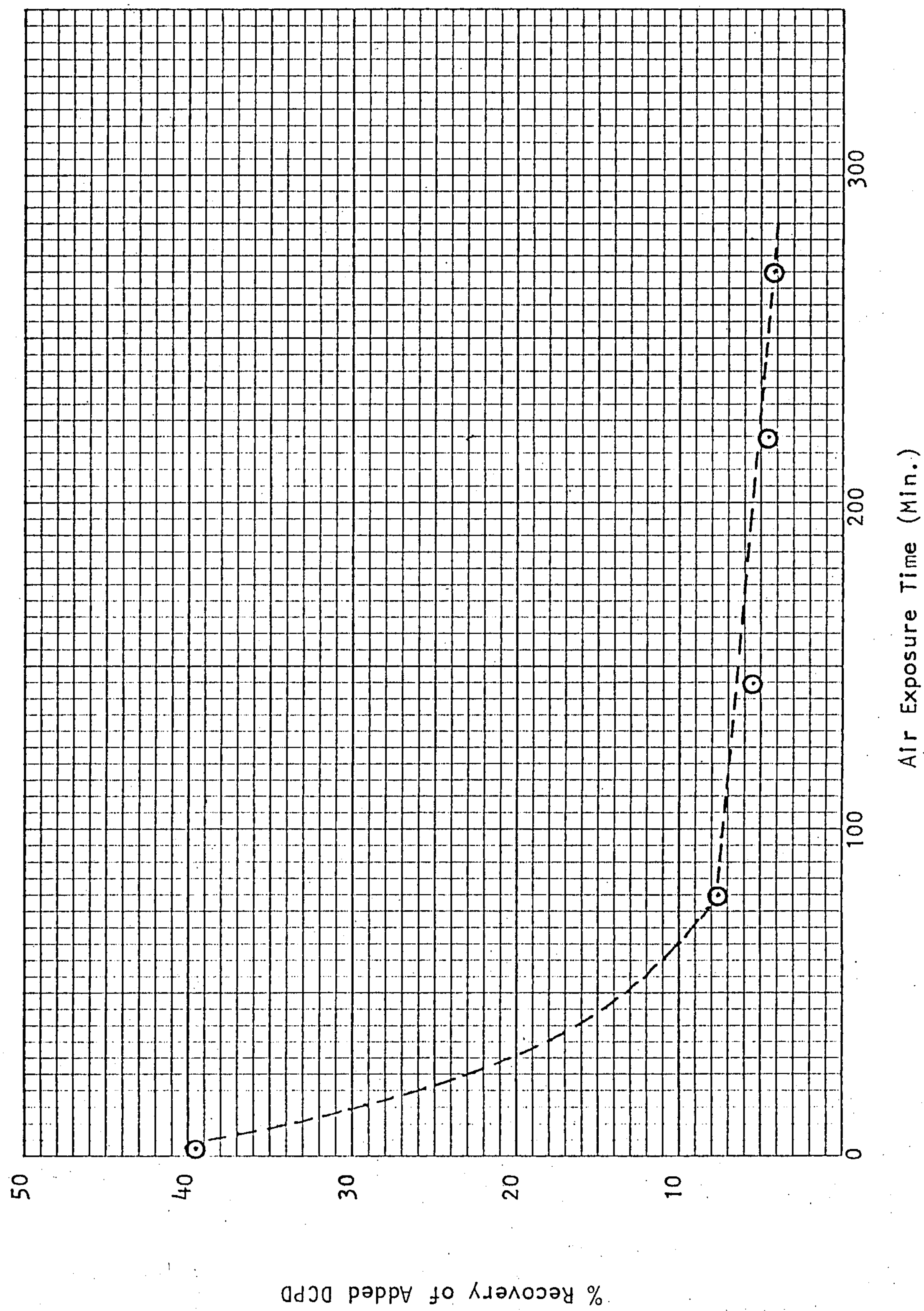


Figure 5. Evaporation Curve for DCPD from Fullerton Soil

- Develop DCPD analysis procedures for soils.
- Continue treatment and analysis of lysimeter soil and water samples.
- Continue gross range finding effectiveness level experiments for DIMP and DCPD in soils.